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# Simultaneous application of ultrasounds and firming agents to improve the quality properties of osmotic + freeze-dried foods

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## ABSTRACT

Dried foods generally show poor rehydration ability and changes in the product properties. In this work, strawberries, used as a model food, were processed using osmotic dehydration (OD) followed by freeze drying (FD) to investigate the effects on the sample colour, texture, microstructure and rehydration. For the first time, a new approach was implemented by adding firming agents (FA) in the osmotic solution to strengthen the cell walls and applying ultrasounds (US) during the pre-treatment to enhance the process mass transfer. FA and US have been implemented often separately but never combined. The resulting samples were then further dried using FD. This strategy revealed to be successful in improving the properties of dried foods compared to FD solely: rehydration capacity was enhanced; colour was better retained, showing colour coefficients closer to the fresh fruit; texture was largely improved, exhibiting the same mechanical properties of the raw material; microstructure was well preserved.

## 1. Introduction

Drying is one of the main products processing in the food industry. It inhibits the microbial spoilage and the enzyme activity, thus extending the product shelf life (de Bruijn et al., 2016). Dried products are more convenient since their low volume allows reducing the packaging, transport and storage costs (Brown, Fryer Norton, Bakalís, & Bridson, 2008).

One of the key parameters that quantify the quality of a dried product is its rehydration capacity, i.e. the ability to reacquire the initial amount of water within its structure. Generally, dried products show moderate or low rehydration capacity, since cellular and structural ruptures occur during the drying process (Vega-Gálvez et al., 2015).

Among the drying techniques, freeze-drying (FD) gained interest since it provides both high water desorption and good retention of the food characteristics (Karam, Petit, Zimmer, Baudelaire Djantou, & Scher, 2016; Shishegarha, Makhlof, & Ratti, 2002). Long process times and high-energy demands, however, are required to obtain safe products, characterised by moisture content (MC) lower than 20–25 g/100 g and water activity ( $a_w$ ) lower than 0.6. These conditions are generally regarded as the threshold values to avoid bacteria proliferation and enzymatic activity that can cause degradation of the product (Ratti, 2001; Stevenson et al., 2015; de Bruijn et al., 2016).

In order to overcome these limitations, some pre-treatments can be

applied, for instance osmotic dehydration (OD). OD is a low-cost method, which allows more colour, aroma, nutritional constituents and flavour retention (Sagar & Suresh Kumar, 2010; Yadav & Singh, 2014). The application of osmotic dehydration allows an intermediate moisture product to be produced, which can be dried further using a conventional technique, with a reduced processing time (da Costa Ribeiro, Aguiar-Oliveira, & Maldonado, 2016; Prosapio & Norton, 2017; Ruiz-López, Huerta-Mora, Vivar-Vera, Martínez-Sánchez, & Herman-Lara, 2010). In a recent paper Prosapio and Norton (2017) investigated the influence of osmotic dehydration on freeze drying performance. They studied the effect of OD operating parameters (type of osmotic agent, temperature, concentration and processing time) and FD processing time on water activity, moisture content, solid gain, texture and rehydration. They showed that the application of the pre-treatment with Fructose 60 °Bx, at 50 °C and 180 min followed by 7-h freeze drying allowed to obtain the same samples' final water activity and moisture content of 18-h freeze drying alone. Nevertheless, they noted that, at the process conditions investigated, the rehydration capacity was lower than that obtained for freeze drying, as previously reported by (Ciurzyńska & Lenart, 2012; Seguí, Fito, & Fito, 2013). In their paper, Prosapio and Norton hypothesized that the cause of the lower rehydration capacity was related to the higher shrinkage that the samples experienced during osmotic dehydration.

Another common pre-treatment in food drying involves the use of ultrasounds (US). This technology has gained interest in recent years as

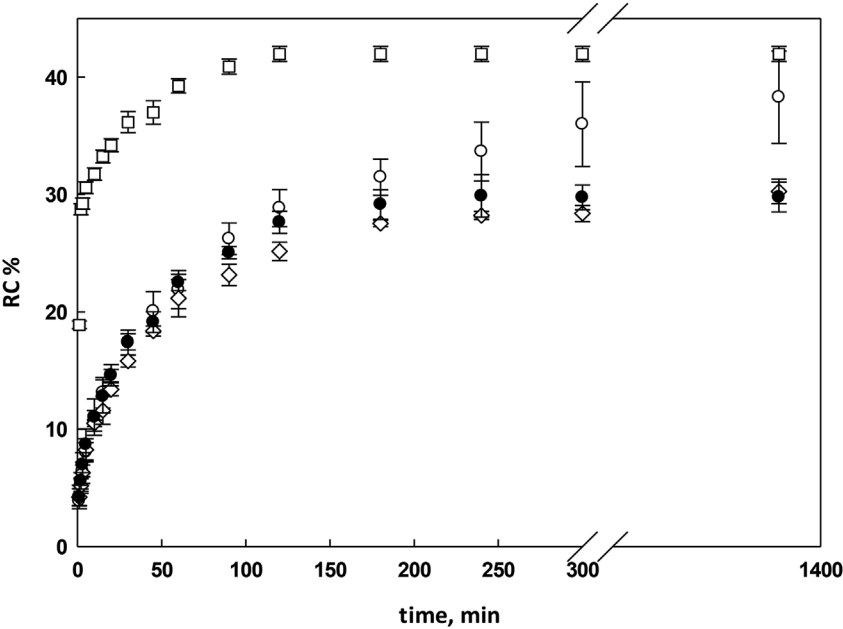
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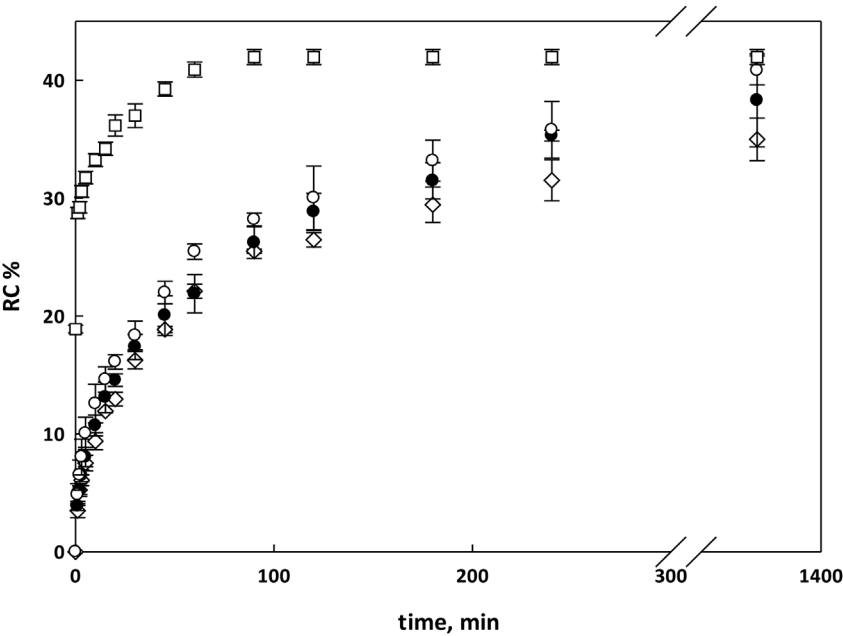
**Table 1**  
OD + FD experiments. Each value is expressed as mean  $\pm$  standard deviation (n = 3).

#	Firming agent	C <sub>FA</sub> [%w/w]	C <sub>OS</sub> [°Bx]	a <sub>w</sub>	MC [g/100 g]	RC %
1	–	–	40	0.439 $\pm$ 0.098 <sup>a</sup>	9.55 $\pm$ 0.75 <sup>a</sup>	24.88 $\pm$ 3.12 <sup>a</sup>
2			50	0.302 $\pm$ 0.074 <sup>a,b</sup>	8.98 $\pm$ 2.82 <sup>a</sup>	36.55 $\pm$ 1.02 <sup>b</sup>
3			60	0.195 $\pm$ 0.006 <sup>b</sup>	7.52 $\pm$ 0.79 <sup>a</sup>	30.00 $\pm$ 2.04 <sup>a,b</sup>
4	Calcium chloride	1	50	0.429 $\pm$ 0.018 <sup>a</sup>	13.56 $\pm$ 1.25 <sup>a,b</sup>	38.30 $\pm$ 3.93 <sup>b</sup>
5		5		0.471 $\pm$ 0.040 <sup>a,c</sup>	20.91 $\pm$ 1.08 <sup>b</sup>	30.06 $\pm$ 1.04 <sup>a</sup>
6		10		0.485 $\pm$ 0.011 <sup>a,c</sup>	31.31 $\pm$ 3.14 <sup>c</sup>	29.78 $\pm$ 1.27 <sup>a</sup>
7	Calcium lactate	1	50	0.429 $\pm$ 0.007 <sup>a</sup>	12.00 $\pm$ 2.38 <sup>a</sup>	40.84 $\pm$ 1.23 <sup>b</sup>
8		5		0.585 $\pm$ 0.064 <sup>c</sup>	17.89 $\pm$ 1.71 <sup>b</sup>	38.13 $\pm$ 1.35 <sup>b</sup>
9		10		0.597 $\pm$ 0.015 <sup>c</sup>	19.83 $\pm$ 4.38 <sup>b</sup>	31.09 $\pm$ 1.10 <sup>a,b</sup>

C<sub>FA</sub>: concentration of firming agent; C<sub>OS</sub>: concentration of osmotic solution; a<sub>w</sub>: water activity; MC: moisture content; RC: rehydration capacity. The values followed by the same letter (abc) in the columns are not significantly different according to one-way ANOVA and Tukey's multiple comparison tests.



**Fig. 1.** Effect of the concentration of calcium chloride (runs #4–6):  $\square$  FD (data taken from (Prosapio & Norton, 2017)),  $\circ$  OD+FD calcium chloride 1% w/w,  $\bullet$  OD+FD calcium chloride 5% w/w,  $\diamond$  OD+FD calcium chloride 10% w/w.



**Fig. 2.** Influence of the firming agent on the rehydration capacity of osmotic + freeze dried samples (runs #2, 4, 7 in Table 1):  $\square$  FD,  $\circ$  OD+FD 1% calcium lactate,  $\bullet$  OD+FD 1% calcium chloride,  $\diamond$  OD+FD.

**Table 2**  
USOD + FD experiments. Each value is expressed as mean  $\pm$  standard deviation ( $n = 3$ ).

#	Firming agent	$t_{OD}$ [min]	$a_w$	MC [g/100 g]	RC %
10	Calcium	15	$0.698 \pm 0.139^a$	$41.35 \pm 11.39^a$	n.p.
11	chloride	30	$0.655 \pm 0.119^a$	$30.77 \pm 5.73^b$	n.p.
12		60	$0.504 \pm 0.040^b$	$23.23 \pm 8.54^b$	$41.59 \pm 1.91^a$
13		90	$0.473 \pm 0.026^b$	$18.97 \pm 4.99^c$	$44.86 \pm 4.34^a$
14		180	$0.412 \pm 0.068^b$	$17.38 \pm 0.94^c$	$36.60 \pm 6.15^b$
15	Calcium	15	$0.686 \pm 0.102^a$	$27.87 \pm 1.93^b$	n.p.
16	lactate	30	$0.421 \pm 0.014^b$	$20.14 \pm 2.49^c$	$49.74 \pm 1.40^c$
17		60	$0.395 \pm 0.009^a$	$18.76 \pm 2.14^b$	$39.23 \pm 2.57^b$
18		90	$0.328 \pm 0.039^a$	$12.91 \pm 4.98^b$	$32.07 \pm 4.66^b$
19		180	$0.285 \pm 0.074^a$	$10.01 \pm 7.33^a$	$29.45 \pm 1.59^b$

$t_{OD}$ : osmotic dehydration time; n.p.: not performed. The values followed by the same letter (abc) in the columns are not significantly different according to one-way ANOVA and Tukey's multiple comparison tests.

it is considered non-thermal and sustainable (Gamboa-Santos, Montilla, Cárcel, Villamiel, & Garcia-Perez, 2014). It has been frequently observed that the implementation of US during OD allows a reduction of the processing time (Garcia-Noguera et al., 2010) and the solid intake (Amami et al., 2017; Barman & Badwaik, 2017; Farhaninejad, Fathi, Shahedi, & Sadeghi, 2017), and a better preservation of the product colour (Garcia-Noguera, Oliveira, Weller, Rodrigues, & Fernandes, 2014). However, it has also been observed that the use of US affects the food tissue, with cellular breakdown and formation of micro-channels, which cause the collapse of the microstructure (Fernandes, Gallão, & Rodrigues, 2009; Rastogi, 2011).

In order to strengthen the structure of dried foods firming agents can be used. Among them, calcium salts have been widely used in food processing as fortifiers. Calcium can interact with the free carboxyl groups of the pectin chains in the cell walls, forming the calcium pectate, a cross-bridge that reduces cell separation and, therefore, the tissue softening. In literature it has been observed that the addition of calcium salts during OD produces a firmer structure (Guiamba, Ahrné, Khan, & Svanberg, 2016; Quiles et al., 2004; Siramard & Charoenrein, 2014) and a good antimicrobial effect (Pereira, Carmello-Guerreiro, Junqueira, Ferrari, & Hubinger, 2010).

Many papers have been published on US-assisted OD and the use of firming agents in the osmotic solution, but no study has been carried

**Table 3**  
Degree of porosity in dried strawberry. Each value is expressed as mean  $\pm$  standard deviation ( $n = 3$ ).

Sample	Porosity %
FD	$80.90 \pm 3.56^a$
Run #2	$41.07 \pm 11.58^b$
Run #7	$57.52 \pm 6.76^c$
Run #16	$61.25 \pm 0.03^c$

The values followed by the same letter (abc) in the columns are not significantly different according to one-way ANOVA and Tukey's multiple comparison tests.

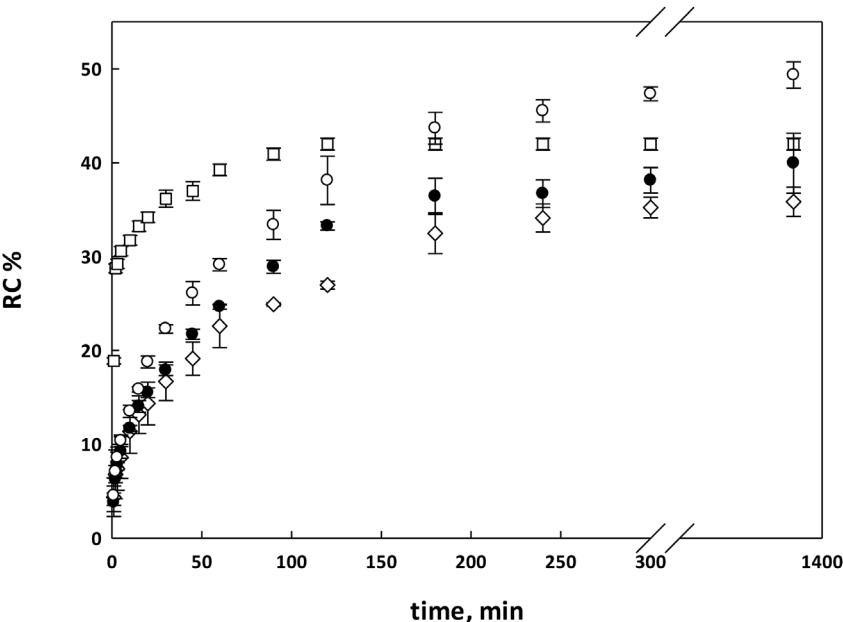
out so far on the combination of them with the aim of optimising the drying process while improving the rehydration and retaining the properties of the fresh products.

In this work, for the first time, calcium chloride and calcium lactate have been employed during ultrasound-assisted osmotic dehydration followed by freeze-drying. The effects of the kind and concentration of firming agent and the sonication time have been investigated to identify the conditions that allow minimising the process time and the damages to the food microstructure. Strawberry has been used as model food, using previous results as a reference (Prosapio & Norton, 2017).

## 2. Materials and methods

### 2.1. Materials

Fresh strawberries (Malling centenary, on average 4 cm in diameter and 7 cm in length) were purchased by a local supermarket and stored in a refrigerator at 5 °C before being used for the experiments. After washing in tap water, draining with blotting paper and removing the external impurities, strawberries (10 g for each experiment) were cut into cubes of 1 cm<sup>3</sup>. Fructose (purity  $\geq 99\%$ ) used as osmotic dehydrator was supplied by Sigma Aldrich (UK). Calcium chloride (purity  $\geq 93\%$ ) used as firming agent was supplied by Fisher Scientific (UK). Calcium lactate used as firming agent was supplied by VWR International (UK). All materials were used as received.



**Fig. 3.** Comparison of the RC % trends:  $\square$  FD (data taken from (Prosapio & Norton, 2017)),  $\diamond$  OD + FD,  $\bullet$  OD (with calcium lactate) + FD,  $\circ$  USOD (with calcium lactate) + FD.

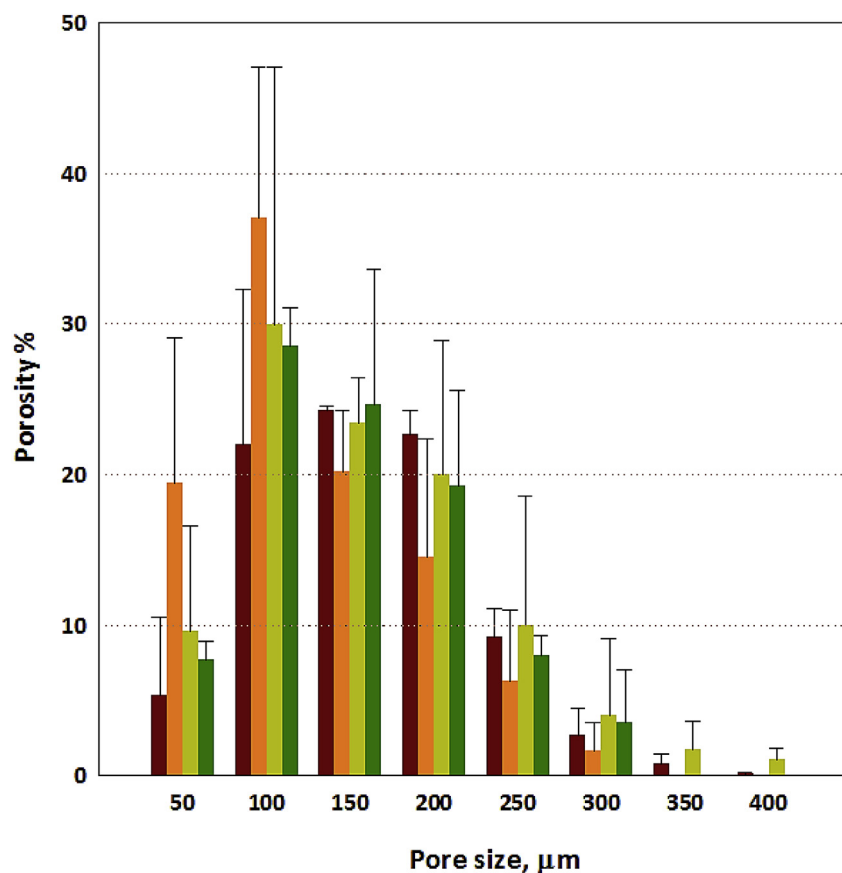


Fig. 4. Comparison of pore size distributions of samples dried using different approaches: (brown) FD, (orange) run#2, (yellow) run#7, (green) run#16. Each value is expressed as mean  $\pm$  standard deviation.

## 2.2. Osmotic dehydration (OD)

Osmotic dehydration experiments were carried out by immersion of 10 g of strawberry cubes into 100 g osmotic solution (formed by 40, 50 or 60 g of fructose dissolved in 60, 50 or 40 g of water to make 40, 50 or 60 °Bx, respectively), under stirring for 180 min at 250 rpm (Stuart, SB162-3, UK). The temperature of the medium was set at 50 °C, in agreement with an optimisation of the process conditions conducted in a previous work (Prosapio & Norton, 2017). The fruit to solution ratio was fixed at 1:10. At the end of each treatment, samples were taken from the beaker and blotted with paper to remove surface water. OD experiments were performed in triplicate for each condition investigated.

## 2.3. Ultrasound-assisted osmotic dehydration (USOD)

USOD experiments were performed by immersion of 10 g of strawberry cubes in a beaker containing 100 g of osmotic + firming agent solution (formed by 50 g of fructose to make 50 °Bx and calcium chloride or calcium lactate 1% w/w dissolved in water, the fruit to solution ratio fixed at 1:10). The beaker was then placed in a sonicating bath (Branson Ultrasonic cleaner 3510, UK) with frequency of 40 kHz, power of 130 W and the temperature of the medium was set and kept constant at 50 °C. The time of ultrasound application was varied from 15 to 180 min. At the end of each experiment, samples were taken and blotted with paper to remove surface water. USOD experiments with calcium chloride or calcium lactate were performed in triplicate for each condition investigated.

## 2.4. Freeze-drying (FD)

Samples were first placed in a freezer at  $-20$  °C for 18 h, applying a freezing rate of around  $0.2$  °C/min, previously measured by use of thermocouples at both sample core and surface. Frozen samples were then lyophilised using a bench top Freeze Dryer (SCANVAC Coolsafe™, model 110-4, Denmark), condenser temperature  $-110$  °C, pressure 0.2 mbar, condition that is defined by the equipment. The processing time was set at 18 h for samples processed only with FD and at 7 h for osmotically pre-treated samples, in agreement with an optimisation of the process conditions performed in a previous work (Prosapio & Norton, 2017). Experiments were performed in triplicate for each condition investigated.

## 2.5. Moisture content analysis

Moisture content (MC) analyses were carried out using a moisture analyser (MB 25, OHAUS, Switzerland). Two grams of sample were placed within the aluminium pans and located over the pan support of the moisture meter. A halogen element inside the moisture meter provides uniform infrared heating. It heats the sample at a set temperature of  $120$  °C until the sample weight becomes constant. Moisture percentage as a function of weight change is recorded and displayed.

## 2.6. Water activity analysis

Water activity ( $a_w$ ) of fresh and dried samples was measured using an AquaLab® dew point water activity meter (model 4 TE, Decagon Devices Inc., Pullman, WA, USA). The temperature controlled sample chamber was set to  $25$  °C.



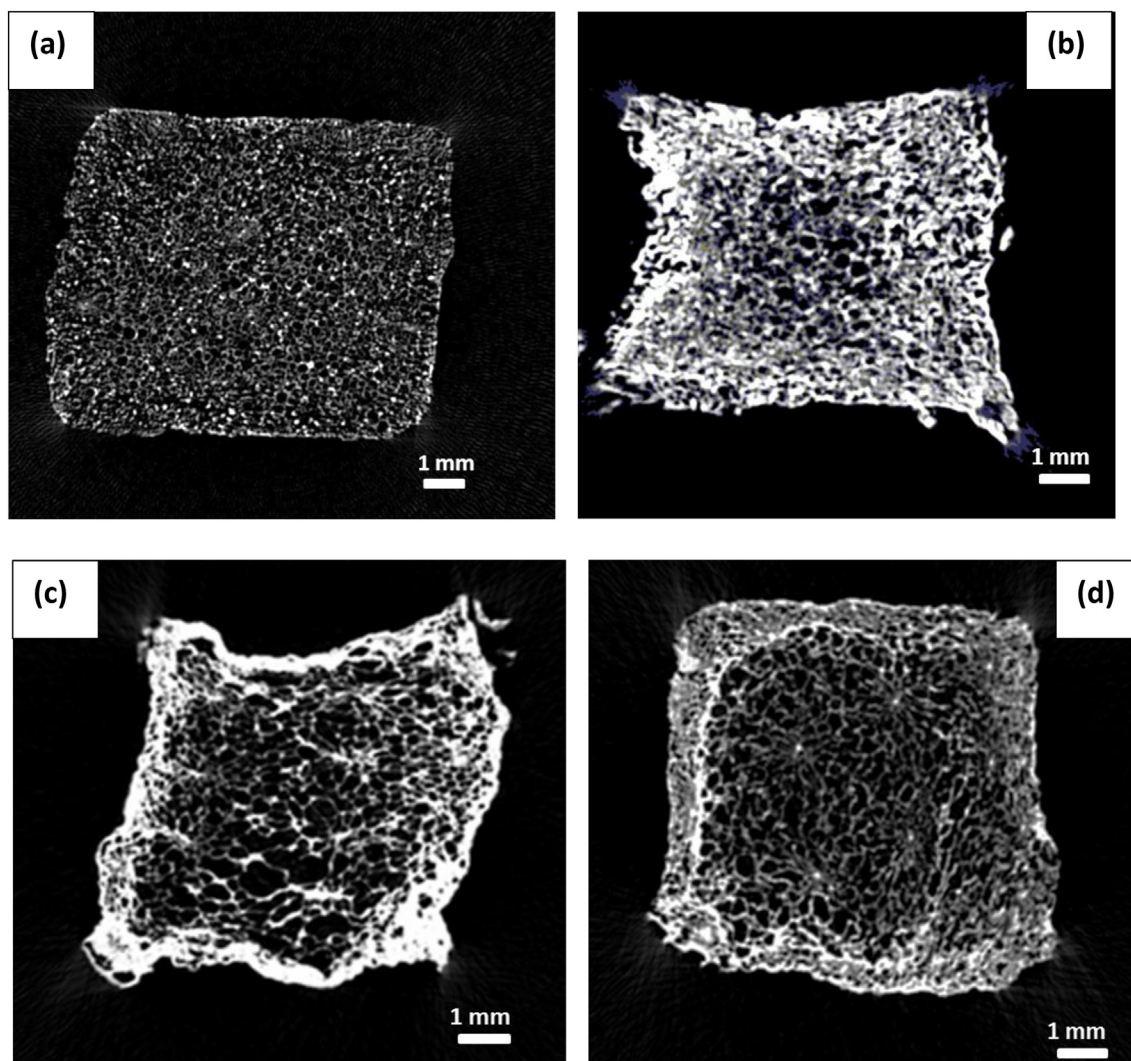


Fig. 5. Micro-CT images of dried samples: (a) FD; (b) OD + FD run#2; (c) OD(FA) + FD run#7; (D) USOD + FD run#16.

Table 4

Colour coefficients of fresh and dried strawberry. Each value is expressed as mean  $\pm$  standard deviation ( $n = 5$ ).

Sample	L*	a*	b*	C <sub>ab</sub> *	$\Delta E$
Fresh strawberry	57.54 $\pm$ 1.79 <sup>a</sup>	65.34 $\pm$ 5.15 <sup>a</sup>	53.48 $\pm$ 8.58 <sup>a</sup>	84.52 $\pm$ 9.09	–
FD strawberry	74.00 $\pm$ 11.35 <sup>b</sup>	32.96 $\pm$ 11.45 <sup>b</sup>	30.06 $\pm$ 7.10 <sup>b</sup>	45.11 $\pm$ 11.20	43.65 $\pm$ 15.71
Run #2	60.00 $\pm$ 14.32 <sup>a,b</sup>	48.70 $\pm$ 15.91 <sup>a,b</sup>	34.81 $\pm$ 12.42 <sup>b,c</sup>	59.91 $\pm$ 19.98	28.77 $\pm$ 19.17
Run #7	59.72 $\pm$ 15.51 <sup>a,b</sup>	47.43 $\pm$ 7.90 <sup>b,c</sup>	36.11 $\pm$ 9.79 <sup>a,b</sup>	58.89 $\pm$ 8.79	29.94 $\pm$ 9.91
Run #16	55.57 $\pm$ 4.04 <sup>a</sup>	58.39 $\pm$ 4.21 <sup>a,c</sup>	44.47 $\pm$ 3.75 <sup>a,c</sup>	73.53 $\pm$ 2.84	12.99 $\pm$ 2.00

L\*: lightness coefficient; a\*: red colour coefficient; b\*: yellow colour coefficient; C<sub>ab</sub>\*: chroma;  $\Delta E$ : relative colour difference index. The values followed by the same letter (abc) in the columns are not significantly different according to one-way ANOVA and Tukey's multiple comparison tests.

## 2.7. Rehydration

Rehydration tests were performed by immersing a weighed amount of dried samples into distilled water at room temperature (samples to water mass ratio 1:100). Samples were removed at regular intervals, blotted with paper to eliminate the surface water and then reweighed. Rehydration capacity (RC %) was measured for all the samples using the following equation:

$$RC = \frac{(w(t) - w_d)}{(w_0 - w_d)} 100 \quad (1)$$

Where:

- $w(t)$  is the sample weight at time  $t$  (g);
- $w_d$  is the dried sample weight (g);
- $w_0$  is the sample initial weight (g).

Then, the rehydration behaviour was determined plotting RC as a function of the time (min). Rehydration tests were performed in triplicate for each treatment.

## 2.8. Microstructure

The structure of dried strawberries was analysed using a Skyscan 1172 X-ray micro-computed tomography ( $\mu$ CT) (Bruker  $\mu$ CT, Belgium) system, with 80 kV maximum X-ray energy, 8 W beam power, 1150 ms

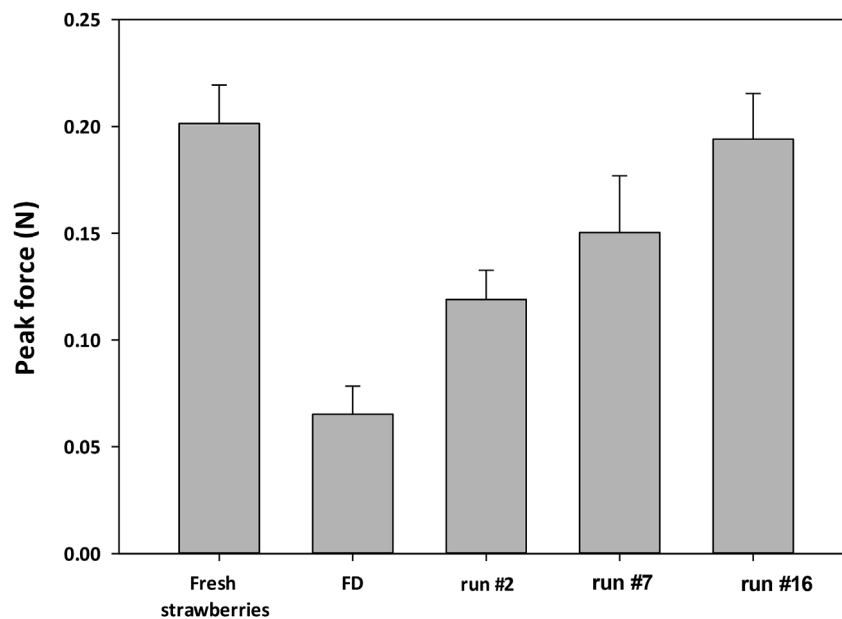


Fig. 6. Puncture penetration testing of strawberries dried using different approaches and rehydrated at room temperature. Each value is expressed as mean  $\pm$  standard deviation.

exposure per projection and  $5.95 \mu\text{m}$  pixel size. Data was reconstructed using NRecon (1.6.10.2, Bruker) and then visualised in 2D and 3D using DataViewer (version 1.5.1.2, Bruker) and CTVOX (version 3.0, Bruker) software, respectively. CTAn (version 1.15.4.0, Bruker) software was thereafter used to determine the porosity of the dried bulk structure and the pore size distribution. For each condition investigated, three samples were analysed.

## 2.9. Colour measurement

The determination of colour was carried out via image analysis, using a methodology adapted from Yam and Papadakis (2004). The external surface colour of fresh and dried samples was evaluated using a high-resolution photographic camera (Canon EOS 5D Mark III) by capturing the images under a proper lighting. The lighting system was formed by two source lamps, positioned on the two sides of a white frame, where the sample was placed, 30 cm above it and at an angle of  $45^\circ$  to the sample plane. The camera was held on a tripod and the lens faced towards the sample. The lens was zoomed to have the whole field of view covered by the sample and focused. Photographs were taken and saved as JPEG files. Colour attributes of samples in each image were measured by Matlab R2016b Image processing software. Images were imported and analysed using an algorithm written in Matlab, which returns the values of the lightness coefficient  $L^*$ , the red colour coefficient  $a^*$  and the yellow colour coefficient  $b^*$ . The colour indicators were then calculated using the following formulas:

$$C_{ab}^* = \sqrt{a^{*2} + b^{*2}} \quad (4)$$

$$\Delta E = \sqrt{(L^{*0} - L^*)^2 + (a^{*0} - a^*)^2 + (b^{*0} - b^*)^2} \quad (5)$$

Where:

- $C_{ab}^*$  is the Chroma;
- $\Delta E$  is the relative colour difference index;
- the subscript 0 is referred to fresh strawberry.

Measurements were carried out analysing 5 samples for each condition investigated.

## 2.10. Texture analysis

A texture analyser (TA.XT plus, Stable Micro System Ltd, UK) with a cylinder probe (2 mm diameter) was used for puncture penetration tests. The probe was used to measure the maximum force required to penetrate an individual rehydrated piece of strawberry, to a depth of 2 mm, positioned horizontally over a heavy-duty platform. The speed of approach of the probe was 2 mm/s and a 5 kg load cell was used. For each experiment, the mean maximum penetration force (N) was recorded. Measurements were carried out analysing 10 samples for each condition investigated.

## 2.11. Statistical analysis

All experiments and measurements were performed in triplicate and reported as mean and standard deviation. Data were analysed by one-way analysis of variance (ANOVA) and Tukey's multiple comparison tests, using SigmaPlot 12.5 Statistical Software. The level of significance was defined as  $p \leq 0.05$ .

## 3. Results and discussion

Fresh strawberries were characterised by initial moisture content equal to  $91.00 \pm 0.05 \text{ g/100 g}$  and water activity equal to  $0.982 \pm 0.004$ . In order to investigate the properties of the dried samples, two set of experiments were performed carrying out OD under stirring (section 3.1) and under ultrasounds (section 3.2).

### 3.1. Osmotic dehydration + freeze-drying (OD + FD)

Experiments were performed using osmotic dehydration (OD) under stirring prior to freeze-drying (FD). The following effects were investigated: the osmotic solution concentration ( $C_{OS}$ ), the firming agent (FA) and its concentration ( $C_{FA}$ ). Table 1 shows a list of the performed experiments, with the indication of the operating conditions employed, the percentage of moisture content (MC) in the final samples (after FD), their water activity ( $a_w$ ) and rehydration capacity (RC %) after 24 h.

The effect of the osmotic solution concentration was investigated at 40, 50 and 60 °Bx (runs #1–3 in Table 1). As observed from the data reported in Table 1, an increase of the sugar concentration resulted in a

decrease of the sample moisture content and water activity, especially for 60 °Bx. However, a different trend was observed for the rehydration capacity, which increased to  $36.55 \pm 1.02\%$  when the sugar concentration was set at 50 °Bx and then decreased at the higher concentration ( $30.00 \pm 2.04\%$ ). These results suggest that higher the driving force, higher the sample shrinkage which, in conjunction with the higher sugar impregnation, limits the rehydration process. Therefore, in order to maximise the rehydration capacity, a concentration equal to 50 °Bx was fixed for the following experiments.

The effect of different concentrations of FA used (1, 5, 10% w/w) was investigated (Table 1). For both salts it was observed that increasing their concentration in the osmotic solution, the water activity and moisture content increased, whereas the rehydration ability decreased, as already reported by (Udomkun, Mahayothee, Nagle, & Müller, 2014). This evidence is probably related both to the formation of the calcium pectate that, at high concentration, may obstruct the mass transfer, and the binding between pectin and water, which forms more viscous solutions (Albano & Nicoletti, 2018). Fig. 1 shows the effect of the calcium chloride concentration on the final rehydration ability and compares it with the RC% of freeze-dried samples. At the lowest concentration of firming agent, the samples'  $a_w$  and MC were below the safety thresholds (0.6 and 20 g/100 g, respectively) and rehydration capacity was higher than that obtained in absence of the firming agent (as shown in Fig. 2). From Fig. 2, it can be observed that the use of calcium lactate results in a slightly higher RC than calcium chloride, but it is still lower than the rehydration exhibited by freeze-dried samples. Despite the formation of the calcium pectate, osmotically pre-treated samples still experienced some shrinkage, as shown later in Fig. 5c. Therefore, the effect of combining ultrasounds (US) and firming agent during the pre-treatment was investigated.

### 3.2. Ultrasound-assisted osmotic dehydration + freeze-drying (USOD + FD)

The osmotic solution used for these experiments was fructose 50 °Bx and 1% w/w firming agent at 50 °C. The effect of the osmotic dehydration time ( $t_{OD}$ ) was investigated (Table 2). Rehydration tests were not performed (n.p.) when the water activity of the sample was higher than 0.6 as they were not safe from bacteria proliferation. As shown in Table 2, when samples were processed using calcium chloride (runs #10–14) over 180 min,  $a_w$  and MC decreased progressively. In terms of rehydration, the highest value ( $44.86 \pm 4.34\%$ ) was observed in correspondence of a processing time equal to 90 min. The reason of this trend is that samples processed for 180 min are stressed for too long, causing cell disruption, microstructure collapse and, therefore, a decrease in the rehydration ability. In the case of calcium lactate (runs #15–19 in Table 2), the trend was similar, but the highest RC ( $49.74 \pm 1.40\%$ ) was obtained using a processing time equal to 30 min. This result allows a reduction of the OD processing time of 83.3% (from 180 to 30 min) and, therefore a substantial decrease of the energy consumption (lower running costs) and the possibility to process more products in the same time period.

As observed from the results reported in Tables 1 and 2, the simultaneous use of ultrasounds and calcium lactate as firming agent increases the rehydration capacity of the dried samples, achieving about 50%. Fig. 3 reports a comparison among the RC curves of freeze-dried samples (data taken from (Prosapio & Norton, 2017)), OD + FD samples (run #2 in Table 1), OD (with FA) + FD samples (run #7 in Table 1) and USOD + FD (run #16 in Table 2). At short rehydration times (up to 120 min), freeze-dried samples rehydrate faster since the structure did not shrink during drying. When OD is applied, the samples undergo shrinkage. For this reason, they require more time to swell and absorb water. However, after 120 min, samples osmotically pre-treated with US and calcium lactate showed the highest RC.

### 3.3. Microstructure

During the freezing step, ice crystals are formed, which leave pores on sublimation. The degree of porosity and the pore size distribution affect the rate and the extent of rehydration.

Samples previously discussed (FD and runs #2, 7, 16) were analysed using X-ray micro-computed tomography. Porosity data are reported in Table 3, whereas the pore size distributions and a cross-section of the dried samples are reported in Figs. 4 and 5, respectively.

Table 3 shows that the freeze-dried samples had the highest porosity, which explains the faster rehydration rate at short time (Fig. 3), with mean pore size around 100  $\mu\text{m}$  (Fig. 4). When osmotic dehydration is applied prior to freeze-drying, a partial collapse of the structure occurs, as shown in Fig. 5b in which white zones, characterised by high density of cell wall membrane, and shape distortion can be observed. As a consequence, porosity was significantly lower (about half of FD samples), with consequent reduced rehydration ability, as previously shown in Fig. 3. The addition of the firming agent contributed to preserve the integrity of the cell walls, thus reducing the collapse. In effect, porosity increased by 57% and larger pores are visible in Fig. 5c. The application of ultrasounds for 30 min during OD gave a porosity of 61% and pores of about 150  $\mu\text{m}$ . This last observation helps explaining the rehydration trends of Fig. 3: at short times, USOD samples rehydrate slower than freeze-dried samples because of the lower porosity; however, at longer times the presence of larger pores allows the absorption of a higher amount of water within the network. In addition Fig. 5d shows that the reduced exposure to heat reduced the collapse of the structure, as dense areas into the samples are less evident, and the shape was more preserved.

### 3.4. Colour

Colour is one of the main quality attributes that influence the product acceptance by the consumer (Holzwarth, Korhummel, Carle, & Kammerer, 2012; Kalt, 2005). During drying, colour changes not only due to water removal, but also because some chemical and biochemical reactions take place, such as Maillard reactions, enzymatic browning, oxidation and some heat-sensitive compounds may be destroyed by thermal exposure (Fellows, 2009; Hii & Law, 2010; Rahman, 2007).

In Table 4, the colour coefficients, measured following the method described in Section 2.10, were reported for fresh strawberries and samples dried with freeze-drying (Prosapio & Norton, 2017), osmotic dehydration + freeze-drying (run #2), osmotic dehydration (with calcium Lactate) + freeze-drying (run #7) and ultrasound assisted osmotic dehydration (with calcium Lactate) + freeze-drying (run #16).

The data in Table 4 shows that drying caused alteration change of the colour for all samples. Freeze-drying had the biggest effect: the lightness coefficient value  $L^*$  was increased whereas the red and yellow coefficients were reduced. Consequently, the colour difference index ( $\Delta E$ ) is the highest among the investigated samples (43.65). The application of the osmotic pre-treatment prior to freeze-drying (run #2) retained more the colour (lower  $\Delta E$ ); this result is in agreement with the literature (Ciurzyńska, Lenart, & Gręda, 2014; de Bruijn & Bórquez, 2014). The use of the firming agent (run #7) had no effect on the colour of dried strawberries, since all the parameters were essentially the same as observed for samples processed without calcium lactate. When osmotic drying was performed with ultrasounds, the colour indicators are the closest to the original colour of fresh strawberries and  $\Delta E$  has the lowest value (about 13.00). This result is probably due to the reduced heat exposure during osmotic drying (30 min instead of 180 min).

### 3.5. Texture

Texture is another important parameter for food acceptability, especially for dried products. During drying, the food tissues are subjected to stress, which causes cracks and deformations, with consequent



softening of the tissues (Lewicki & Pawlak, 2003).

Fig. 6 shows the bar chart with the results of the puncture tests for fresh strawberries and rehydrated samples processed with FD, OD + FD (run #2), OD (with calcium Lactate) + FD (run #7) and USOD (with calcium Lactate) + FD (run #16).

As reported in a previous work (Prosapio & Norton, 2017), freeze-drying causes a large reduction in firmness. This result can be explained by the formation of large crystals during the freezing step. These crystals would be the responsible of the breaking the cells, resulting in a weaker structure. The application of osmotic dehydration allows more retention of the structure and caused less effect, as shown in Fig. 4. This can be explained as at intermediate moisture less ice is formed. In addition, the presence of sugar will result in smaller crystals since it favours the nucleation kinetic rather than the crystal growth (Petzold & Aguilera, 2009). When calcium lactate is added to the osmotic solution, the textural properties were further retained since, as mentioned in the Introduction, the formation of the calcium pectate increases the pectin retention (Mauro et al., 2016). The highest firmness preservation was observed for samples treated with ultrasounds for 30 min during OD. In this case, the force required to puncture the sample is comparable to that of the fresh material (Fig. 6). In fact, the reduced exposure time to heat (30 min instead of 180 min) combined with the use of the firming agent allows more preservation of the fruit texture than conventional osmotic dehydration.

#### 4. Conclusions

In this work, it was shown that the simultaneous application of ultrasound and firming agent during osmotic dehydration prior to freeze-drying is a promising strategy to improve the quality properties of dried foods: rehydration capacity was enhanced as compared to freeze-dried samples; colour was better retained, showing colour coefficients closer to the fresh strawberry; texture was largely improved, exhibiting the same mechanical properties of the fresh fruit; the fruit shape was maintained as the microstructure was well preserved and the cell walls were not damaged. Moreover, it was possible to shorten the osmotic dehydration process time, resulting in a potential reduction of the energy costs.

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#### References

- Albano, K. M., & Nicoletti, V. R. (2018). Ultrasound impact on whey protein concentrate-pectin complexes and in the O/W emulsions with low oil soybean content stabilization. *Ultrasonics Sonochemistry*, 41, 562–571. <https://doi.org/10.1016/j.ultsonch.2017.10.018>.
- Amami, E., Khezami, W., Mezrigui, S., Badwaik, L. S., Bejar, A. K., Perez, C. T., et al. (2017). Effect of ultrasound-assisted osmotic dehydration pretreatment on the convective drying of strawberry. *Ultrasonics Sonochemistry*, 36, 286–300. <https://doi.org/10.1016/j.ultsonch.2016.12.007>.
- Barman, N., & Badwaik, L. S. (2017). Effect of ultrasound and centrifugal force on carambola (Averrhoa carambola L.) slices during osmotic dehydration. *Ultrasonics Sonochemistry*, 34, 37–44. <https://doi.org/10.1016/j.ultsonch.2016.05.014>.
- Brown, Z. K., Fryer, P. J., Norton, I. T., Bakalis, S., & Bridson, R. H. (2008). Drying of foods using supercritical carbon dioxide — investigations with carrot. *Innovative Food Science & Emerging Technologies*, 9(3), 280–289. <http://dx.doi.org/10.1016/j.ifset.2007.07.003>.
- de Bruijn, J., & Bórquez, R. (2014). Quality retention in strawberries dried by emerging dehydration methods. *Food Research International*, 63(Part A), 42–48. <http://dx.doi.org/10.1016/j.foodres.2014.03.029>.
- de Bruijn, J., Rivas, F., Rodriguez, Y., Loyola, C., Flores, A., Melin, P., et al. (2016). Effect of vacuum microwave drying on the quality and storage stability of strawberries. *Journal of Food Processing and Preservation*, 40, 1104–1115. <http://dx.doi.org/10.1111/jfpp.12691>.
- Ciurzyńska, & Lenart, A. (2012). Erratum: Rehydration and sorption properties of osmotically pretreated freeze-dried strawberries (Journal of Food Engineering). *Journal of Food Engineering*, 113, 361. <http://dx.doi.org/10.1016/j.jfoodeng.2012.06.014>.
- Ciurzyńska, A., Lenart, A., & Greda, K. J. (2014). Effect of pre-treatment conditions on content and activity of water and colour of freeze-dried pumpkin. *Lebensmittel-Wissenschaft und -Technologie- Food Science and Technology*, 59(2, Part 1), 1075–1081. <http://dx.doi.org/10.1016/j.lwt.2014.06.035>.
- da Costa Ribeiro, A. S., Aguiar-Oliveira, E., & Maldonado, R. R. (2016). Optimization of osmotic dehydration of pear followed by conventional drying and their sensory quality. *Lebensmittel-Wissenschaft und -Technologie- Food Science and Technology*, 72, 407–415. <http://dx.doi.org/10.1016/j.lwt.2016.04.062>.
- Farhaninejad, Z., Fathi, M., Shahedi, M., & Sadeghi, M. (2017). Osmotic dehydration of banana slices using direct and indirect Sonication: Optimization and microstructure analysis. *Journal of Food Process Engineering*, 40(1), <http://dx.doi.org/10.1111/jfpe.12336> e12336-n/a.
- Fellows, P. J. (2009). *Food processing technology: Principles and practice*. Elsevier.
- Fernandes, F. A. N., Gallão, M. I., & Rodrigues, S. (2009). Effect of osmosis and ultrasound on pineapple cell tissue structure during dehydration. *Journal of Food Engineering*, 90(2), 186–190. <https://doi.org/10.1016/j.jfoodeng.2008.06.021>.
- Gamboa-Santos, J., Montilla, A., Cárcel, J. A., Villamiel, M., & Garcia-Perez, J. V. (2014). Air-borne ultrasound application in the convective drying of strawberry. *Journal of Food Engineering*, 128, 132–139. <https://doi.org/10.1016/j.jfoodeng.2013.12.021>.
- Garcia-Noguera, J., Oliveira, F. I. P., Gallão, M. I., Weller, C. L., Rodrigues, S., & Fernandes, F. A. N. (2010). Ultrasound-assisted osmotic dehydration of Strawberries: Effect of pretreatment time and ultrasonic frequency. *Drying Technology*, 28(2), 294–303. <http://dx.doi.org/10.1080/07373930903530402>.
- Garcia-Noguera, J., Oliveira, F. I. P., Weller, C. L., Rodrigues, S., & Fernandes, F. A. N. (2014). Effect of ultrasonic and osmotic dehydration pre-treatments on the colour of freeze dried strawberries. *Journal of Food Science & Technology*, 51(9), 2222–2227. <http://dx.doi.org/10.1007/s13197-012-0724-x>.
- Guimamba, I., Ahrné, L., Khan, M. A. M., & Svanberg, U. (2016). Retention of  $\beta$ -carotene and vitamin C in dried mango osmotically pretreated with osmotic solutions containing calcium or ascorbic acid. *Food and Bioprocess Processing*, 98, 320–326. <https://doi.org/10.1016/j.fbp.2016.02.010>.
- Hii, C., & Law, C. (2010). Product quality evolution during drying of foods, vegetables and fruits. *Drying of foods, vegetables and fruits*, 1, 125–144.
- Holzwarth, M., Korhummel, S., Carle, R., & Kammerer, D. R. (2012). Evaluation of the effects of different freezing and thawing methods on color, polyphenol and ascorbic acid retention in strawberries (Fragaria  $\times$  ananassa Duch.). *Food Research International*, 48(1), 241–248. <https://doi.org/10.1016/j.foodres.2012.04.004>.
- Kalt, W. (2005). Effects of production and processing factors on major fruit and vegetable antioxidants. *Journal of Food Science*, 70(1).
- Karam, M. C., Petit, J., Zimmer, D., Baudelaire Djantou, E., & Scher, J. (2016). Effects of drying and grinding in production of fruit and vegetable powders: A review. *Journal of Food Engineering*, 188, 32–49. <http://dx.doi.org/10.1016/j.jfoodeng.2016.05.001>.
- Lewicki, P. P., & Pawlak, G. (2003). Effect of drying on microstructure of plant tissue. *Drying Technology*, 21(4), 657–683. <http://dx.doi.org/10.1081/DRT-120019057>.
- Mauro, M. A., Dellarosa, N., Tylewicz, U., Tappi, S., Laghi, L., Rocculi, P., et al. (2016). Calcium and ascorbic acid affect cellular structure and water mobility in apple tissue during osmotic dehydration in sucrose solutions. *Food Chemistry*, 195, 19–28. <https://doi.org/10.1016/j.foodchem.2015.04.096>.
- Pereira, L. M., Carmello-Guerreiro, S. M., Junqueira, V. C. A., Ferrari, C. C., & Hubinger, M. D. (2010). Calcium lactate effect on the shelf life of osmotically dehydrated guavas. *Journal of Food Science*, 75(9), E612–E619. <http://dx.doi.org/10.1111/j.1750-3841.2010.01847.x>.
- Petzold, G., & Aguilera, J. M. (2009). Ice Morphology: Fundamentals and technological applications in foods. *Food Biophysics*, 4(4), 378–396. <http://dx.doi.org/10.1007/s11483-009-9136-5>.
- Prosapio, V., & Norton, I. (2017). Influence of osmotic dehydration pre-treatment on oven drying and freeze drying performance. *Lebensmittel-Wissenschaft und -Technologie- Food Science and Technology*, 80, 401–408. <http://dx.doi.org/10.1016/j.lwt.2017.03.012>.
- Quiles, A., Hernando, I., Pérez-Munuera, I., Llorca, E., Larrea, V., & Ángeles Lluç, M. (2004). The effect of calcium and cellular permeabilization on the structure of the parenchyma of osmotic dehydrated 'Granny Smith' apple. *Journal of the Science of Food and Agriculture*, 84(13), 1765–1770. <http://dx.doi.org/10.1002/jsfa.1884>.
- Rahman, M. S. (2007). *Handbook of food preservation*. CRC press.
- Rastogi, N. K. (2011). Opportunities and challenges in application of ultrasound in food processing. *Critical Reviews in Food Science and Nutrition*, 51(8), 705–722. <http://dx.doi.org/10.1080/10408391003770583>.
- Ratti, C. (2001). Hot air and freeze-drying of high-value foods: A review. *Journal of Food Engineering*, 49, 311–319. [http://dx.doi.org/10.1016/S0260-8774\(00\)00228-4](http://dx.doi.org/10.1016/S0260-8774(00)00228-4).
- Ruiz-López, I. I., Huerta-Mora, I. R., Vivas-Vera, M. A., Martínez-Sánchez, C. E., & Herman-Lara, E. (2010). Effect of osmotic dehydration on air-drying characteristics of chayote. *Drying Technology*, 28, 1201–1212. <http://dx.doi.org/10.1080/07373937.2010.482716>.
- Sagar, V. R., & Suresh Kumar, P. (2010). Recent advances in drying and dehydration of fruits and vegetables: A review. *Journal of Food Science & Technology*, 47(1), 15–26. <http://dx.doi.org/10.1007/s13197-010-0010-8>.
- Seguí, L., Fito, P. J., & Fito, P. (2013). A study on the rehydration ability of isolated apple cells after osmotic dehydration treatments. *Journal of Food Engineering*, 115, 145–153. <https://doi.org/10.1016/j.jfoodeng.2012.08.038>.
- Shishhegarha, F., Makhlouf, J., & Ratti, C. (2002). Freeze-drying characteristics of strawberries. *Drying Technology*, 20(1), 131–145. <http://dx.doi.org/10.1081/DRT-120001370>.
- Siramard, S., & Charoenrein, S. (2014). Effect of ripening stage and infusion with calcium lactate and sucrose on the quality and microstructure of frozen mango. *International Journal of Food Science and Technology*, 49(9), 2136–2141. <http://dx.doi.org/10.1111/ijfs.12553>.

- Stevenson, A., Cray, J. A., Williams, J. P., Santos, R., Sahay, R., Neuenkirchen, N., et al. (2015). Is there a common water-activity limit for the three domains of life? *The ISME Journal*, 9, 1333–1351. <http://dx.doi.org/10.1038/ismej.2014.219>.
- Udomkun, P., Mahayothee, B., Nagle, M., & Müller, J. (2014). Effects of calcium chloride and calcium lactate applications with osmotic pretreatment on physicochemical aspects and consumer acceptances of dried papaya. *International Journal of Food Science and Technology*, 49(4), 1122–1131. <http://dx.doi.org/10.1111/ijfs.12408>.
- Vega-Gálvez, A., Zura-Bravo, L., Lemus-Mondaca, R., Martínez-Monzó, J., Quispe-Fuentes, I., Puente, L., et al. (2015). Influence of drying temperature on dietary fibre, rehydration properties, texture and microstructure of cape gooseberry (*Physalis peruviana* L.). *Journal of Food Science & Technology*, 52, 2304–2311. <http://dx.doi.org/10.1007/s13197-013-1235-0>.
- Yadav, A. K., & Singh, S. V. (2014). Osmotic dehydration of fruits and vegetables: A review. *Journal of Food Science & Technology*, 51(9), 1654–1673. <http://dx.doi.org/10.1007/s13197-012-0659-2>.
- Yam, K. L., & Papadakis, S. E. (2004). A simple digital imaging method for measuring and analyzing color of food surfaces. *Journal of Food Engineering*, 61(1), 137–142. [https://doi.org/10.1016/S0260-8774\(03\)00195-X](https://doi.org/10.1016/S0260-8774(03)00195-X).